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IS 3504 (2006): Thymol [PCD 18: Natural and Synthetic Fragrance Materials]

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भारतीय मानक

थाइमोल — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

THYMOL — SPECIFICATION

(Third Revision)

ICS 71.100.60

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2006

Price Group 2

FOREWORD

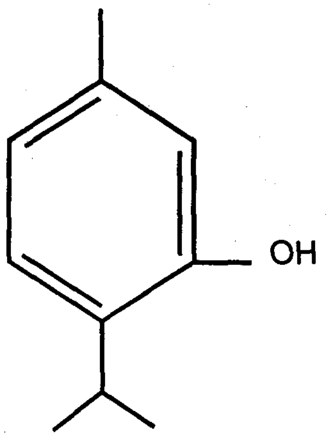
This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Fragrance Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first issued in 1965 and subsequently revised in 1975 and 1992. First revision of the standard was carried out to align with trade practices prevalent in the perfumery industry at that time.

In the second revision, requirement for freezing point was deleted and in place of melting point, requirement for melting range was prescribed. Besides, requirement for residue on evaporation was amended.

In this revision, requirement of residue on evaporation has been made stringent.

Thymol ($C_{10}H_{14}O$) is a crystalline phenol. It is also known as 5-methyl-2-*iso*-propyl-phenol. It is used as an antiseptic and preservative. It is represented by the following structural formula:



5-methyl-2-*iso*-propyl-phenol
(Molecular mass 150)

Synthetic thymol is now being manufactured on large scale indigenously from Delta-3-carene and also from menthones as derived from dementholized oil.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

THYMOL — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for thymol, synthetic.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 4) : 2005/ ISO 592 : 1998	Determination of optical rotation (*third revision*)
(Part 6) : 2005/ ISO 875 : 1999	Determination of solubility in ethanol (*third revision*)
(Part 10) : 2005/ ISO 4715 : 1978	Determination of residue on evaporation (*third revision*)
(Part 16) : 1989	Determination of melting point and melting range (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 2001	Glossary of terms relating to fragrance and flavour industry (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, the definitions given IS 6597 shall apply.

4 REQUIREMENTS

4.1 Description

4.1.1 The material shall be in the form of colourless or translucent, monoclinic or hexagonal crystals. It shall have a pungent spicy odour reminiscent of *AJOWAN*. When placed on the tongue, it shall impart a burning sensation.

4.1.2 The material shall be examined for its colour as prescribed in IS 326 (Part 2).

4.1.3 The material shall sink in cold water and when the temperature is raised to about 45°C, it shall melt and rise to the surface.

4.1.4 A solution of the material in ethanol (90 percent, *v/v*) shall be optically inactive [*see* IS 326 (Part 4)].

4.1.5 Take 1 g of the material in a test tube, add 5 ml of 10 percent (*m/v*) solution of sodium hydroxide and heat in a water-bath. A clear, colourless or pale-red solution shall be formed, which shall become darker on standing without the separation of any oily drops. On adding a few drops of chloroform and agitating the mixture, a violet colour shall be produced.

4.1.6 Dissolve a small crystal of the material in 1 ml of glacial acetic acid, add 6 drops of concentrated sulphuric acid and one drop of nitric acid; the liquid shall exhibit a deep bluish-green colour when viewed against reflected light.

4.2 Solubility

One gram of the material shall be soluble in 3 ml (80 percent by volume) of ethanol when tested as prescribed in IS 326 (Part 6) and freely soluble in ether and chloroform.

4.3 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in airtight containers, permitting a minimum of air space, as agreed to between the purchaser and the supplier.

5.1.1 Direct contact of the material with the galvanized or mild steel containers shall be avoided.

5.1.2 The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

Each container so packed shall bear legibly the following information:

a) Name of the material;
b) Indication of source of manufacture;
c) Batch number and date of manufacturing; and
d) Net and gross mass.

5.2.1 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

6.2 Number of Tests

All the characteristics given shall be tested on the composite sample.

7 TESTS METHODS

7.1 Tests shall be conducted as prescribed in col 4 and col 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements of Thymol
(*Clause* 4.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			IS No.	Annex
(1)	(2)	(3)	(4)	(5)
i)	Colour[1]	Colourless or translucent	326 (Part 2)	—
ii)	Odour	Pungent spicy, thyme like	2284	—
iii)	Residue on evaporation, percent	0.05	326 (Part 10)	—
iv)	Melting range, °C	49 - 51	326 (Part 16)	—
v)	Thymol, percent by mass by GC, *Min*	99	—	Annex A

[1] Thymol being a phenol, darkens on storage and also when comes in contact with air and sunlight.

ANNEX A

[*Table* 1, *Clause* 4.3, *Sl No.* (v)]

GAS CHROMATOGRAPHIC ANALYSIS OF THYMOL

A-1 GENERAL

A-1.1 The chromatographic conditions given here are for guidance only.

A-1.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, cyclohexane, hexane or acetone) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column. The detector signals, on transmission to the recorder, plot the chart. From the specific area under various peaks corresponding to the specific constituents, the quantities of different constituents are determined.

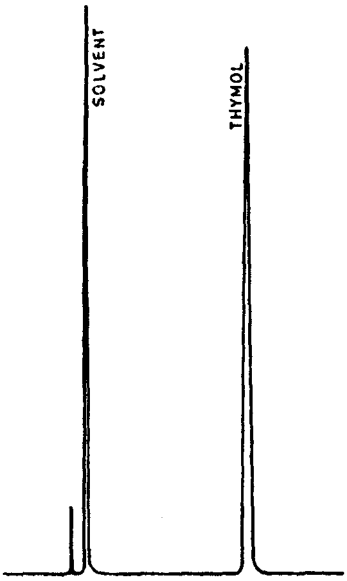


FIG. 1 A TYPICAL CHROMATOGRAM FOR THYMOL

Sample	*Thymol*
a) *Column:*	
Material	Stainless steel
Length	3 m
Orifice	0.32 cm
Stationary phase and solid support	FFAP[1] 10 percent by mass chromosorb WHP 100-120 mesh
b) Carrier gas	Nitrogen
Flow rate	20 ml/min
c) *Conditions:*	
Column temperature	200°C
Injection port temperature	210°C
d) *Detector:*	
Type	FID
Temperature	230°C

[1] Free fatty acid phase in carbowax 20 M treated with nitrophthalic acid.

A-2 APPARATUS

Any suitable gas chromatograph and column capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for thymol with the following chromatographic conditions is shown in Fig. 1:

A-3 CALCULATION

Peak areas are calculated either by the most commonly used triangular method or automated integration. When an electronic integrator is used, concentrations of the constituents on the basis of the peak areas on chromatogram are automatically calculated and presented as a printout. For specific constituents, internal standard method is employed for higher accuracy.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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